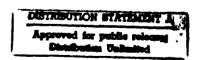
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ENVIRONMENTAL DEGRADATION OF HIGH TEMPERATURE COMPOSITES

R. C. Cochran T. M. Donnellan R. E. Trabocco Naval Air Development Center Warminster PA 18974 USA

> J. Thompson Naval Air Systems Command Washington DC 20361 USA



#### Summary

A study was performed to assess the effect of galvanic corrosion phenomena on the strength of graphite/bismaleimide(BMI) composites. The results indicate that degradation ocurred in BMI composites galvanically coupled to aluminum alloys. The mechanism responsible for the degradation involves hydroxyl ion generation in the cathodic reaction. Optical and electron microscopy of the surface of coupled specimens showed a great deal of cracking and deterioration of the resin. This phenomenon is thought to be associated with stresses in the resin imposed by thermal or chemical processes These cracks may be an indication that the mechanism of degradation is not simply the hydrox1 attack on the resin but a combination of chemical and mechanical attack. Composite-aluminum material couples were exposed to salt water/fuel solutions and to salt spray environment and then composite properties were determined. Conventional protection schemes were evaluated. The results indicated that the performance retention was test specific and that bearing tests were most sensitive to the galvanically induced degradation. Significant bearing strength loss was found when the protection schemes failed. In addition, a systematic study of the electrochemical conditions which were most important in control of the degradation rates was performed.

## Introduction

High temperature composites offer significant weight savings over conventional materials such as steel and titanium. I mide based composites are the only organic matrix materials that have been demongtrated for applications in the 149°C (300°F) to 288°C (550°F) region. The chemical structure of the resin determines the material operating temperature. Polymers based on bismaleimide(BMI) and condensation polyimide chemistries (figure 1) have been developed for engine and airframe components. In many cases the imide based composite is in direct contact with steel or sluminum. In a recent laboratory test program, graphite/bismaleimide composites immersed in salt water while in contact with a metal were observed to degrade. This degradation

resulted in removal of resin and delamination of the composite within several days.

Because of the importance of polyimide composites for Navy applications a program was initiated at NADC to examine the degradation phenomena. The Navy's efforts have focussed on evaluation of the severity of the degradation in existing and emerging aircraft, development of a methodology to mitigate the phenomenon and on identification of the degradation mechanisms. The program was performed in three tasks, specifically salt fog exposure, cleaning fluid exposure and electrochemistry. Salt fog and salt water exposures were used to evaluate the severity of the problem and to evaluate currently used protection schemes. These experiments used specimen designs in which composites were electrically coupled to metal. Electrochemistry experiments were used to assess the importance of specific exposure variables in the degradation process.

## Background

The effect of water on polymer matrix graphite composites in the fleet is a reduction of the thermal stability of the material. This effect is a well characterized reversible plasticization process. Moisture is also known to cause corrosion in metal aircraft components. The Navy spends considerable time and effort on corrosion maintenance of the metal aircraft in the shipboard environment. One form of corrosion is caused by the electrical coupling of dissimilar metals. Galvanic corrosion occurs when there is a difference in the electrical potentials of two conducting materials exposed in a corrosive solution. In the couple, the more corrosion resistant material becomes the cathode where reduction reactions occur while the other more active material becomes the anode where oxidation reactions take place. cathodic reaction that occurs depends on the solution conditions. In neutral or basic solutions, the reaction is the reduction of water and oxygen to produce hydroxyl ions. Graphite is cathodic to aluminum and steel and composites utilizing this type of reinforcement will cause corrosion of these metals when they are

connected electrically. This electrical coupling in structural components is usually accomplished either through the fasteners used to attach composite skins to metal substructure or through adhesive bonds.

Graphite composite driven corrosion of aircraft structural alloys has been studied previously. Fischer and DeLuccia(1) measured a large 1 volt potential difference between graphite/epoxy composites and 7075 aluminum alloys. This potential provided the driving force for extensive corrosion of the alloy. Mechanical testing of the couples indicated that the corrosion was responsible for significant strength loss in the aluminum components. Additional efforts have examined the effectiveness of various protection schemes for the prevention of galvanic corrosion.
Thompson, White and Snide(2) reported that protection schemes which consisted of coatings on the composite and the metal, sealant on the faying surface and around the fastener, and a fiberglass barrier ply dramatically reduced the corrosion found in unprotected couples. These authors cautioned that the protection schemes were only effective if all surfaces were coated and if the sealant layers were not porous.
Similarly, DeLuccia and Brown(3) found that isolation coatings significantly reduced the galvanic corrosion induced fatigue strength loss in aluminum alloys. In all of the work described in the literature there was no indication of degradation of the epoxy resins used in the composite.

The degradation of imide polymers through hydrolysis by moisture or bases has been studied by a number of workers. Strong bases attack the imide group in the polymer structure and produce amic acid salts and amines figure 2. The most widely studied material has been Kapton wire insulation. Environmental factors such as temperature, stress, moisture concentration and basicity have all been found to affect degradation rates. The actual mechanism responsible for the degradation observed has also been addressed. Askins(4) modelled the degradation phenomena with an arhennius relationship and further found that the strength loss in the material followed an exponential decay, first order reaction type response. Wolf and Soloman(5) showed that under specific exposure conditions, hydrolysis rates could be significantly reduced through annealing of the polymer prior to exposure. These authors speculated that the degradation could be a mechanochemical process.

## Discussion of Experimental Results

Evaluation of Galvanic Degradation Processes

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Exposure Specimen Study. The initial effort in the program was the verification of the results reported regarding the degradation phenomena. A set of specimena was fabricated with U.

S. Polymeric AS-4/V-378A coupons connected to 2024 T-6 aluminum coupons of the same dimensions. The specimen size used was 7.5 cm x 1.25 cm x .2 cm (8 ply quasifsotropic laminate). The composite and Al were tied together with a nylon string. Three samples were placed in glass jars containing various combinations of JP-5 (jet fuel) and 3.5% NaCl solution. The combinations included a 50/50 mix of JP-5 and neutral salt water, a 50/50 mix of JP-5 and pH 2 salt water and a jar half filled with neutral salt water. A forth jar with a 50/50 mix of salt water and JP-5 was made up with only the composite sample in it. These jars were placed in an oven at 82°C (180°F). The jars were removed at 24 hour intervals and the samples were observed. The total exposure period was four days.

The first observation regarding these exposed samples was that there was degradation of the composite in a short period of time. The type of attack varied depending on the nature of the solution. Specimens in water/fuel solutions showed an effect which was concentrated in the interface between the fluids. Over a period of time, the affected zone grew up the edge and in towards the center of the specimen. Specimens half immersed in water showed a more general attack over the portion of the specimen above the water surface. All of the degradation occurred on the surface opposite the mating surface of the specimens. The degradation was evidenced first by a lightening of the surface and then exposure of bare fibers.

The sample exposed to the pH 2 solution showed similar degradation to the pH 7 exposed sample. These pH 2 specimens were not expected to degrade in the same manner as those exposed to neutral water since the nature of the reduction reactions in acid and neutral solutions are different and also because the low pH solution was expected to react with any hydroxl ions. A measurement of the pH of this salt solution showed that it had increased to 4.8 during the exposure time. The pH increased due to the production of aluminum hydroxide. These observations indicate that hydroxyl attack is not the only important feature of the degradation phenomena. It also shows the importance of the local solution chemistry i.e.; the chemistry of the environment where degradation occurs. In the samples exposed to only salt water, the degradation did not appear to be as severe, however it covered a much larger area than the fuel water samples. The degradation was not visible until 48 hours of exposure.

The second observation made was that the aluminum attached to the degraded composite specimens had considerable surface corrosion. The corrosion appeared to be qualitatively similar to that reported by Fischer and De Luccia(1).

After 4 days of exposure the samples were removed from the solutions and inspected with optical and scanning electron microscopy(SEM). Examination of the water/fuel exposed specimens showed that the resin had been completely removed from the fibers on the surface in the area just above the interface. The resin higher up the specimens in the fuel phase but still in the affected zone was cracked and fibers were visible below the surface. SEM micrographs from three areas are shown in figure 3. The specimens exposed to only water had much less exposed fiber, however the cracks and chipped resin were more visible over The resin had been thinned the surface. down a great deal on these specimens. SEMs of the surface of the water fuel samples showed that the resin was heavily cracked above the degraded area, figure 3A. Moving down the specimen a large number of rough chips were observed the surface of the specimen, figures 3B. Finally in the area of greatest degradation there were fibers with a thin resin coating, bare fibers and chips of resin visible, figure 3C.

The information obtained from these exposure tests does not allow quantitative analysis of the degradation mechanism to be provided. insights into the mechanism that can be stated based on the observations. The degradation of the coupled specimens was affected by the formation of hydroxyl ions at the cathode in the galvanic cell In the specimens exposed to only salt water the thin layer of water that condensed on the surface of the specimen above the water level was the electrolyte. This thin layer of water provided an area for the reaction to proceed and allowed the hydroxide ions to concentrate on the surface. In the area below the water the hydroxide ions were able to diffuse away from the surface and no degradation was observed. The samples exposed to jet fuel/salt water mixtures showed a concentrated degradation. effect was due to a concentration of hydroxyl ions and oxygen in the meniscus between the water and fuel. The hydroxyl lons are nearly insoluble in the fuel and when formed diffuse down to the interface. The fact that the scid water solution caused similar degradation to the neutral solution indicates that the degradation process is driven by local chemical environments.

Evaluation of Protection Schemes. The next set of experiments performed were designed to evaluate the effectiveness of currently used protection methods for graphite/epoxymetal couples for the prevention of BMI composite degradation. The procedure is outlined in MIL-F-7179. The protection schemes proposed for the graphite/BMI composites consist of a layer of fiberglass/BMI cocured on the surface of the composite and epoxy primer on the mating surface of the laminate. A sealant is applied to the mating surface and around any fasteners in the structure. The aluminum in contact with

graphite reinforced composites is anodized and primed with epoxy primer. There were three screening studies performed which examined the effect of specific protection schemes on composite strength.

The first set of specimens fabricated were used to screen paints and sealants for their ability to protect the composite under aggressive exposure conditions. Four primers, a topcoat and a one coat primer/topcoat were evaluated. The coatings evaluated included; MIL-P-23377 solvent based epoxy primer, MIL-P-85582 water based epoxy primer, Koroflex flexible epoxy primer, a high temperature urethane primer Desoto 825-009, MIL-C-85285 urethane topcoat and Unicoat, a self priming top coat recently developed at NADC. The composite specimens were ply (+,-,0,0,+,-,90)s IM7/5250-4. The aluminum was chromate conversion coated 2024-T6. Composite and Al specimens coated with these materials were cured for one week at room temperature. composite and Al were attached using titanium 6,4 fasteners with A286 nuts. Each coating was evaluated with no sealant, an inhibited sealant and an uninhibited sealant. The coupled specimens were immersed to half height in neutral salt water (3.5% NaCl) and exposed at 82°C (180°F). The specimens were removed from the oven and visually inspected once a day for 7 days. days the specimens were removed for testing. The effects of this exposure on the specimen surface condition are shown in figure 4. The unprotected coupled specimens used as controls completely delaminated after 7 days. The other paint schemes showed various amounts of degradation from none for the urethane primer to severe for the water based The use of top primer and koroflex. coats improved the surface appearance compared to the primer alone. Unicoat provided a high level of protection. One surprising finding was that the sealants did not affect the amount of blistering observed. The primers blistered to the same degree with inhibited and uninhibited sealant. composite specimens were tested in 4 point flexure ASTM D790 at room temperature. The results are given in table 1. These results show that the flexure strength correlated with the amount of degradation observed on the surface. However, the flexure strength of the specimens which had inhibited sealant was higher than those which had uninhibited sealant. An observation of the most severely degraded samples showed that the primer had fibers embedded in it, this was evidence that the primer bonded to the surface however, due to degradation of the resin, fibers and primer were lifted from the surface.

The next set of specimens were fabricated to evaluate the effects of fiberglass surface plies, sealant and high temperature aging of the paint. The samples used for this study were 24 ply (0,90) IM-7/5250-4. The aluminum was .32 cm (.125in.) 2024-16 sheet. The

composite and aluminum specimens were 2.5 cm X 10 cm. (1 X 4 in.). A MIL-S-85430 polysulfide sealant was applied to the faying surface and the fasteners were coated with uninhibited polysulfide sealagt before installation. Two .468 cm (3/16 inch) holes were drilled one half inch from the edges of the specimens. The samples were connected using titanium 6,4 alloy bolts with an A286 alloy nut. The assembled specimen is shown in figure 5. The test matrix used in this evaluation is shown in table 2. The variables examined included the fiberglass ply, the sealant, the type of primer, and the condition of the primer. primer, and the condition of the primer. Specifically, bare and glass covered samples with and without polysulfide sealant with either a solvent based (MIL-P-23377) or a water based primer (MIL-P-8-5582) were evaluated. Some of the coated composite samples were heat aged at 149°C (300°F) for 100 hours before they were assembled. After the sealant had fully cured (1 week) the samples were exposed in a salt spray chamber. Most of the specimens were exposed for 30 days. In addition, another set of specimens which were fabricated with MIL-P-23377 primer, MIL-C-82582 and 825-009 high temperature urathene primer were exposed for 90 days. The exposure conditions for all of the salt fog specimens conform to the ASTM G-84 specification.

After 30 days, the specimens were removed from the salt spray environment and visually inspected. Each set of samples was photographed and then disassembled. After disassembly the composite samples were sectioned in half. The samples were placed in a bearing fixture and tested in compression. This specimen was chosen in order to test the area around the hole. It was expected that this would be the most vulnerable area due to the bare composite and exposed fibers in the hole.

After 30 days of exposure some of the specimens were severely blistered. Samples with water based primer and no sealant had the most severe blistering. Examples of specimens with and without glass and with and without sealant are shown in figure 6. Blisters were visible on all surfaces. The glass scrim ply on the surface had no effect on the number of blisters. The use of a faying surface sealant with water based primer had little effect on the amount of blistering. In some cases the primer flaked off the specimens during exposure and testing. The area under the primer flaked off the specimens during exposure and testing. The area under the primer same observed to be slightly degraded. The pH of the fluid in the blisters was measured to be in the 12 to 14 range. As was noted above, this basic solution will cause damage to the BMI resin. The solvent based primer provided better protection, however blisters occurred near the edge on several specimens. The use of a sealant on the faying surface and glass scrim reduced the number of blisters on the water based primer specimens. The edge blisters in the solvent primer samples are probably due to edge thinning of the primer as it

stretched across the corner of the specimen. The samples that were heat aged for 100 hours at 149 °C (300°F) showed no blisters for either the solvent or water based primers. In order to evaluate the amount of degradation in the hole several specimens were sectioned and microscopically evaluated. The samples exposed in salt spray all show minor amounts of degradation in the hole area. There was a qualitative correlation between the hole quality and the bearing strength of the samples.

Bearing test results are given in table 2. The data show a decrease from the control specimen strength. average for the control was 550 MPa (80 KSI) while the coupled samples ranged from 406 MPa (58.9 KSI) for water based primer, no glass and sealant to 517 MPa (75.0 KSI) for solvent primer, temperature aged, glass surface ply samples. The results show that the glass ply had little effect on the bearing strength of the laminate. In all cases except glass with sealant the solvent borne primer specimens exhibited higher strength. Samples with sealant in the faying surface showed lower properties than the samples without sealant in every case. This result was not expected since the sealant is an added layer protection between the composite and aluminum. It is possible that the sealant acted to concentrate any cathodic reaction on a limited area of the composite.

Specimens that were removed after 90 days showed some blistering and wrinkling of the primer. The glass, primer, sealant combinations are shown in table 3. The amount of degradation on the surface was not significantly different than the 30 day exposure samples. Bearing strength results were found to beslightly lower for the 90 day exposure specimens. The most interesting difference found was that the high temperature primed specimens with sealant had bearing strengths significantly lower than those of unsealed specimens (68.0 KSI vs 91.0 KSI). The control for this set of laminates was 86.0 KSI. This data supports the hypothesis that the sealant acts to concentrate the degradation process at the hole.

The third set of specimens prepared for exposure were open hole compression specimens. These samples consisted of 24 ply (+45, 0, -45, 90), IM-7/5250-connected to 2024 T6 aluminum sample of the same size with a titanium fratener in the center of the specimen. The coatings and isolation schemes used are shown in table 4. Several of the samples were scratched to simulate a flaw that may occur in the protection scheme during service. These specimes, were also exposed in a salt spray environment for 30 days. After exposure, the specimens were disassembled rad were tested at room temperature in an MTS test machine at a rate of .125 cm/min (.05 in/min).

These specimens showed some blistering of the primer. Scratched samples did not show any increased blistering over unscratched samples. Areas under blisters and near the fasteners showed some sign of resin damage however it was limited to the resin on the surface of the composite. The data indicated that there was no significant change in the strength of the material after exposure. Non coupled control samples which were exposed for the same time had an average strength of 410 MPa (59.7 KSI) while the primed specimens which did not have a fiberglass barrier ply had a strength of 400 MPa (57.9 KSI). The use of topcoat and sealant provided a slight improvement in the strength.

The results of this testing indicate that the amount of composite that is effected by the degradation is small for this type of exposure. Specimen designs which test the mechanical integrity of the exposed surfaces are most sensitive to the degradation phenomena. The bearing strength is dependent on the quality of the material in the hole where the degradation process is concentrated. The compression strength was not affected because the small amount of degradation was insignificant in the test geometry.

#### Electrochemistry

In an effort to determine which variables in the process most strongly effect the rate and severity of degradation, a factorial test matrix was developed. An electrochemical cell was used to expose the BMI composite to various environments. The variables evaluated included salt concentration, oxygen concentration, pH, time, current density and temperature. Two levels of each variable were evaluated. The levels of each variable were as follows: Salt concentration .1 and 3.5%, aerated and dearated water, pH 7 and 11, 1 and 4 day exposure, temperatures of 25 C (75 F) and 83 C (180 F) and current densities of 40 and 160 micro amps per square centimeter.
The specimen consisted of a 24 ply 5250-4 composite. Electrical connection was made to the composite by drilling a hole, sanding both surfaces to expose fibers, coating the hole and area around it with silver filled paint and installing a steel nut and bolt with the wire lead. The test area was then masked off and the specimens primed with epoxy primer and topcoated with urethane paint. Three samples were exposed at each condition listed in table 6. The test cell with the specimen is shown in figure 7. The composite specimen was the cathode and platinum mesh electrode the snode This cell allowed the duplication of the reactions in the freely corroding cell. Each test was replicated three times to eliminate extraneous results. During exposure bubbles formed on

During exposure bubbles formed on the surface of the composite and in some specimens cracks and blisters formed as shown in (figure 8). Similar blisters

The state of the s

were observed by Tucker and Brown (6) in graphite/vinyl ester laminates coupled to steel. After removal from the cell some of the specimens with little or no blisters were observed to have turned lighter in color. After exposure the specimens were tested in flexure. The results are given in table 5. The results showed that high current, high temperature and long times had the greatest effect on the specimen appearance and flexure strength. Many blisters and low flexure strengths were observed for specimens exposed under these conditions. Reduction of the data and determination of significant factors was performed on the flexure results. This screening showed that the variables were ranked in the following order; current density, time, temperature, oxygen concentration, pH, salt concentration. The most significant factors were current density, time and temperature. These variables had greater than .95 significance level. There was very little interaction between any of the variables. The lack of significance of the oxygen concentration is an important factor since oxygen is required for the reaction. Further study of this factor will be carried out in future studies.

#### Conclusion

The mechanical property testing performed indicated that bearing strength of BMI compo ites was most sensitive to the degradation caused by galvanic corrosion. Observation of the specimens showed that the damage to the inside of the hole was slight, however the effect on the composite bearing strength can be significant. The degradation which results from the 30 day salt spray exposure appears to only effect the exposed surface of the composite. Therefore, the bearing strength decreased but the open hole compression strength was not affected. Similarly, flexure tests on specimens exposed in the electrochemical cell showed large decreases in strength when the surface ply was blistered. However when the surface was only discolored there was only a minimal loss of strength.

The electrochemical tests point out that temperature, time and current density are important factors in the degradation process. The rate and severity of degradation are greatly effected by these factors while oxygen

and salt concentration had little effect. The Navy has dealt with corrosion and degradation in the past. Methods of preventing corrosion of metals and inspecting for corrosion are part of all aircraft production processes and maintenance manuals. Degradation of imide based composites is potentially rapid and severe. Strict attention must be paid to material selection, application and fleet inspection. Potential primary or critical application of imide composites must consider the associated risk of galvanic degradation vs alternate materials. The risk analysis must also consider the use of

corrosion resistant materials in structural components where the potential exists for a galvanic couple with imide based composites. The degradation of imide composites structures should be addressed through proper material selection, complete galvanic isolation during assembly, and inspection and maintenance in the fleet. However, the inspection rates and the repair processes required for support of these structures are unknown at this time.

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Table 1 Coating/sealant flexure test results

Coating/Sealant	Flexure Strength MPa
23377	1585
85582	1144
Koroflex	1523
Urethane	1781
23377/83430	1635
85582/83430	1583
Koroflex/83430	1648
Urethane/83438	1987
23377/81733	1741
85582/81733	1971
Koroflex/81733	1866
Urethane/81733	1756
23377/85285	2035
85582/85285	2041
Koroflex/85285	1575
Urethane/85285	1924
Unicoat	1894
Control	1753

Table 2: Bearing Results

PROTECTION SCHEME	STRENGTH (MPa)		
	AVG	S	
Water based primer, sealant	406	39.4	
Solvent based primer, sealant	438	30.0	
Water based primer	454	31.7	
Solvent based primer	477	40.5	
Water based primer, sealant, glass	436	38.6	
Solvent based primer, sealant, glass	442	25.0	
Water based primer, temperature aged	498	36.1	
Solvent based primer, temperature aged	517	31.4	
Control	550	32.1	

Table 3 Three month bearing test results.

PROTECTION SCHEME	STRENGTH		
	MPa	S	
High temperature urethane	627	27	
High temperature urethane, sealant, glass	469	10	
Solvent based primer, sealant glass	403	21	
Control	593	24	

Table 4: Open Hole Compression Test Results

Protection Scheme	Strength (MPa)		
	AVG	s	
Bare / Primer / Sealant	399	16.5	
Glass / Primer / Sealant	408	3.3	
Bare / Primer / Topcoat	407	12.6	1
Bare / Primer / Topcoat / Sealant	422	15.1	į
Glass / Primer / Topcoat	398	14.5	i
Glass / Primer / Topcoat / Sealant	420	13.2	į
Glass / Primer / Topcoat / Sealant / Scratched	432	15.1	,
Control	427	18.6	

Table 5 Electrochemical test results

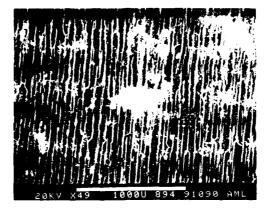
	RUN#	SALT	OXYGEN	рH	TIME	TEMP	CURRENT	FLEXURE;
	%				DAYS	°C	mA/cm2	MPa
	1	.1	YES	7	1	83	40	1489
	2	.1	NO	7	4	83	160	593
	3	3.5	NO	11	4	25	160	855
	4	.1	NO	11	4	25	40	938
	5	3.5	YES	11	4	83	160	676
	6	3.5	YES	7	4	25	40	1241
	7	.1	NO	11	1	83	160	710
	8	3.5	NO	7	4	83	40	986
	9	.1	YES	11	4	83	40	1365
	10	3.5	NO	7	1	25	160	1248
	11	.1	YES	11	1	25	160	1152
	12	3.5	YES	11	1	25	40	1420
	13	3.5	NO	11	1	83	40	1055
	14	.1	YES	7	4	25	160	972
:	15	.1	NO	7	1	25	40	1475
	16	3.5	YES	7	1	83	160	882

4,4 Bismalelmidodipheyimethane

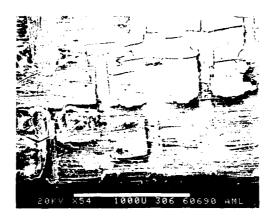
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Figure 1 Chemical structure of bismaleimide and polyimide.

Figure 2 Imide degradation by base.



A Above degradation



B In the degraded area

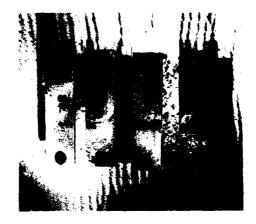


C In the degraced area

Figure 3 Electron micrographs of degraded surface



A Uncoated



B Primer



C Primer and Uninhibited sealant



D Primer and inhibited sealant

Figure 4 Coating evaluation specimens

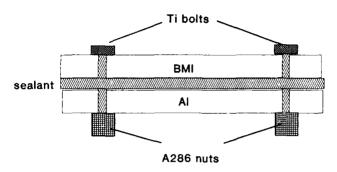
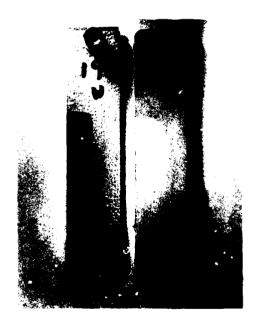


Figure 5 Salt spray exposure specimen







B Glass and sealant

Figure 6 Bearing specimens after 1000 hr salt spray

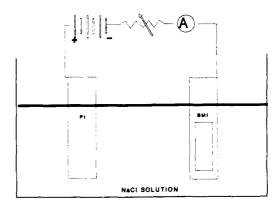


Figure 7 Electrochemical cell

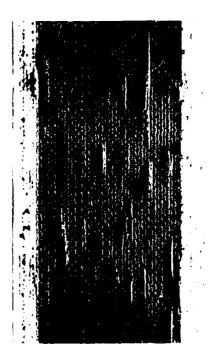


Figure 8 Surface of sample exposed in the electrochemical cell